

## **ASCI $\mu$ P: Fieldable Microsystems**

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### **LONG-TERM GOALS**

The overall goal of this work involves the development of a self contained, fieldable microfluidic chemical sensor fabricated with micromachining techniques. The targeted integrated sensor comprises a microanalytical segment, integrated detection electronics and associated telemetry functionality.

### **OBJECTIVES**

The main focus of the effort is to advance the design, fabrication and field testing of a microfluidic device as an adaptive chemical analyzer that utilizes on-chip reaction separation and either a photonic or an electrochemical detection strategy. The implementation of two different fluid-flow pump mechanisms and the use of hybrid packaging for electronic integration are a portion of the development effort. The scope of work included multiple developments: Field deploy a separations device; advance integration of optical elements on chip; complete lab systems for chip CE and HPLC; Continue the inclusion of on chip detection circuitry; develop a mini high-pressure pump; develop a mini power supply; place temp and conductivity functions on a planar surface.

### **APPROACH**

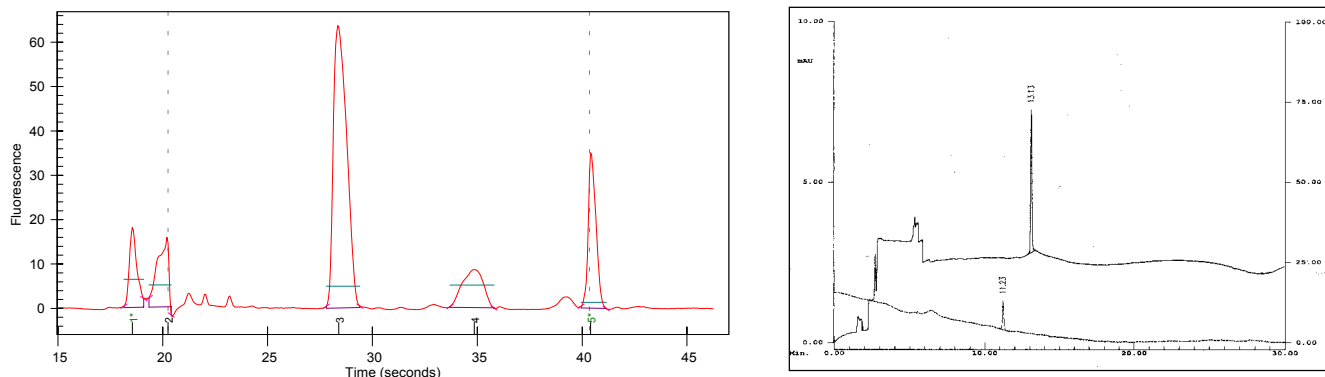
As in the past we rely on the advances in our rapid prototyping fabrication methods for ease of creation of the MEMS and to further develop the fast analyzer/analytical method for the field. The microfluidic chemical sensor has three main paths of development: laser induced fluorescence (LIF) detection; electrochemical detection; and incorporation of wireless technology. In addition two modes of analysis are being developed CE and HPLC. Method development for seawater matrices is a necessary task for field analysis. The current period entails the transfer of the lab based chemical processor for deployment in the field along with physical sensors. Additional microsystems redesigns and subsequent testing and field deployment is expected as follow-on work.

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## WORK COMPLETED

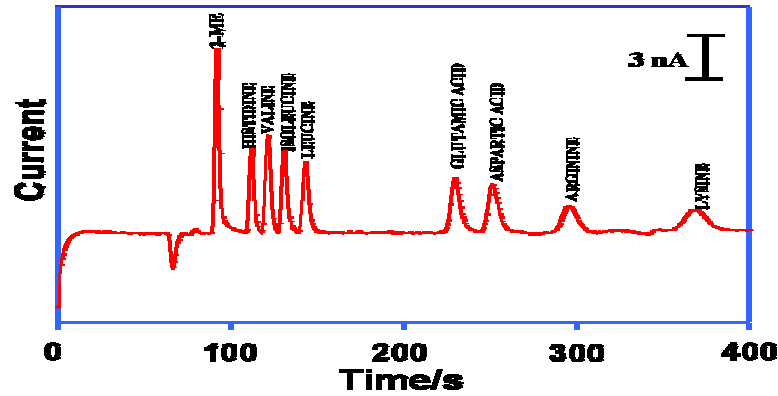
### *Laser induced Fluorescence Microchip*

We have focused the use of the CE-LIF based microchip analyzer (based on a compact 635 nm diode laser excitation and 670 nm emission) on separations for chemical distribution info. Proteins will be our initial targeted field compounds. Figure 1 illustrates the detection of protein distributions in the 15kDa to 200 kDa range on the microchip. We are currently evaluating a simplified C-4 solid phase extraction material for protein preconcentration from seawater for field use and we have developed a unique inline preconcentration step (Figure 1 right). We are schedule for shipboard evaluation in Oct. 2001



***Figure 1. microchip CE-LIF of protein distribution 15kDa- 200kDa range (left); amino acid preconcentration from seawater using sol-gel coated precolumn***

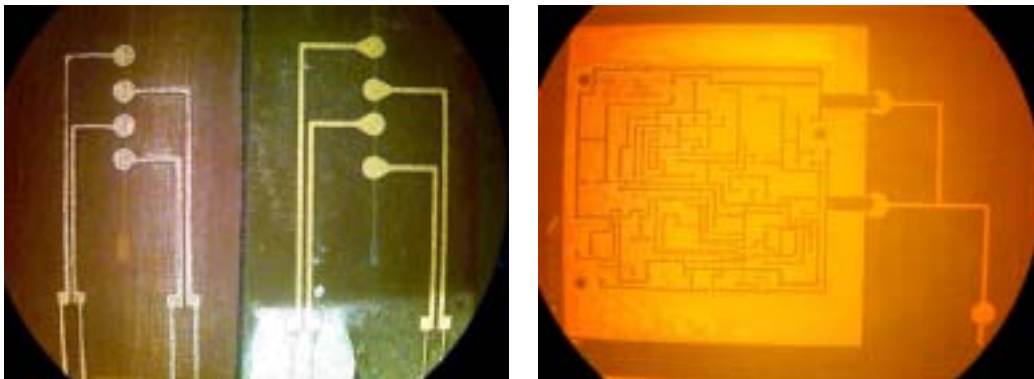
The compact epi-fluorescence detector was the first step in optical bench size reduction. We are testing super bright LED die (~75um sized) for on chip integration. The microoptical elements have integrated Bragg gratings permitting direct attachment to the planar chip with integrated light focusing. We have terminated our 355nm CE-LIF technique using an optical gating scheme. Current progress with reducing the size of short-wave lasers precludes a true miniature system. We have focused separations of amino acid mixtures instead using electrochemical detection of the exact same chemical system, microelectrodes will permit us to achieve a compact system. Figure 2 exhibits detection of an amino acid using electrochemical detection through our collaborator Prof Joe Wang (NMSU). The analysis time is slower than biological response times but we anticipate future time improvements.



**Figure 2. uCZE electrochemical detection of an amino acid using voltammetry of electrodes at the exit of the separation channel**

#### *Microelectronic and Micromechanical Fabrication*

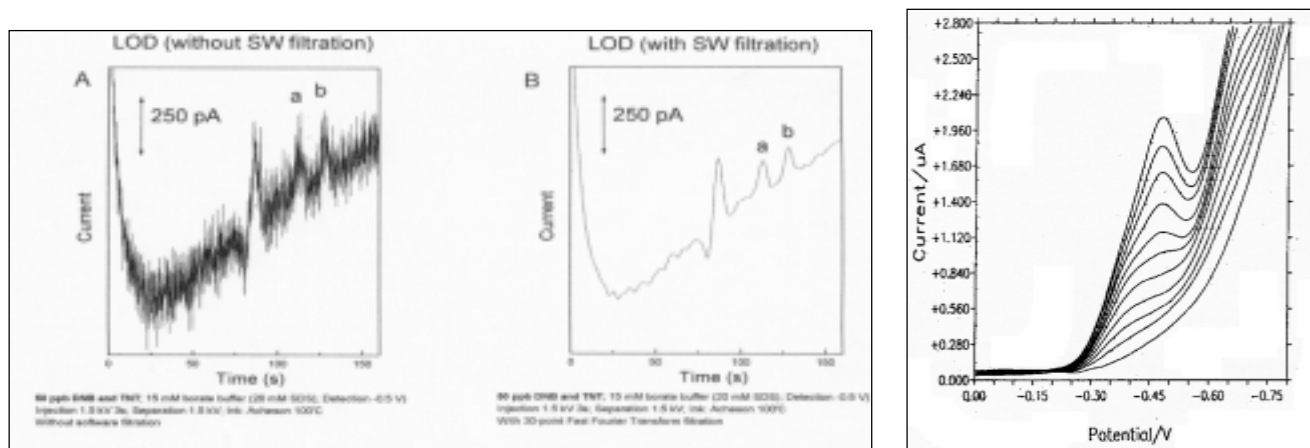
We have continued in creating features for microelectronics and micromechanics with our maskless photolithographic technique for the purpose of microchannel creation and microelectronic integration. Figure 3 shows both a polyimide chip generated using our maskless patterning system and a recently released chip offered commercially from Europe.



**Figure 3. (Left) Optical micrograph of our rapid prototyped integrated microchannel/electrodes (L) versus plasma etched commercial version (R) recently released. (Right) Integrated Microcircuits and microchannel**

## Electrochemical Detection Chip

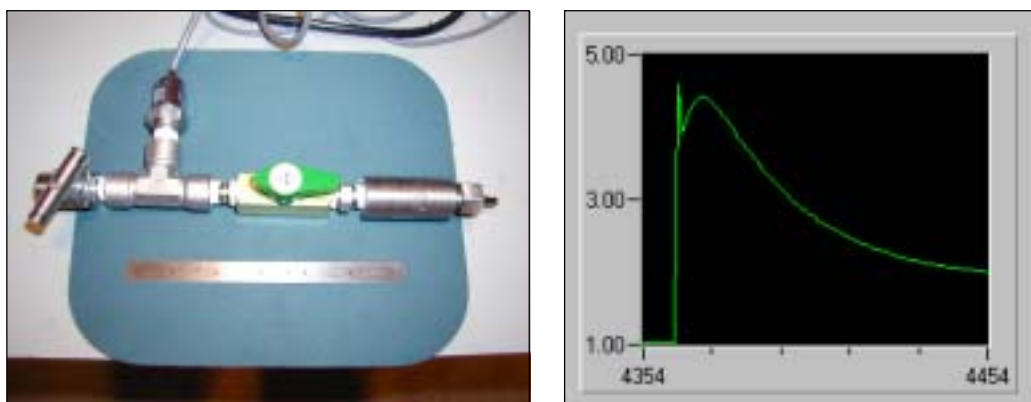
We have made continued progress for the electrochemical detection mode of the CE device and pump based device through our partnership with Dr Wang and focusing effort on explosives detection beyond the amino acids (above).



**Figure 4. (Left) Chip Electropherograms for mixtures containing (a) DNB and (b) TNT at the 50 ppb concentration level. Shown are the raw data (A) and data filtered using 15-point Fast Fourier Transformation filtration (Right) Pump-based carbon fiber remote voltammetric sensor for in-situ monitoring of TNT: square voltammograms for 0.5 NaCl solutions containing increasing levels of TNT in 500 ppb increments.**

## HPLC- pump

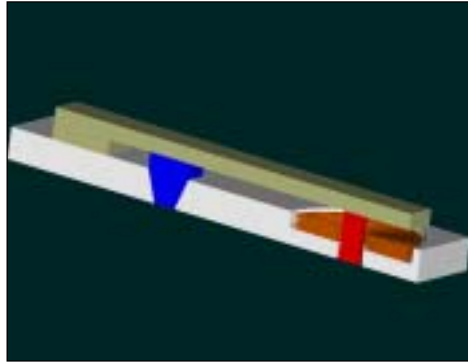
For the HPLC version system we have continued progress on the high pressure fluid micropump. We have tested the concept and we have devised a fabrication process for onchip patterning of the explosive material. We have tested successfully a polymeric explosive (epoxy and perchlorate mix) for onchip photopatterning.



**Figure 5. (Left) High Pressure Pump Prototype ~ 10 ml of capture volume; (Right) Pressure output in psi using (\$0.03) 1.0 grains of medium density ball powder**

### *Micro Power Generation*

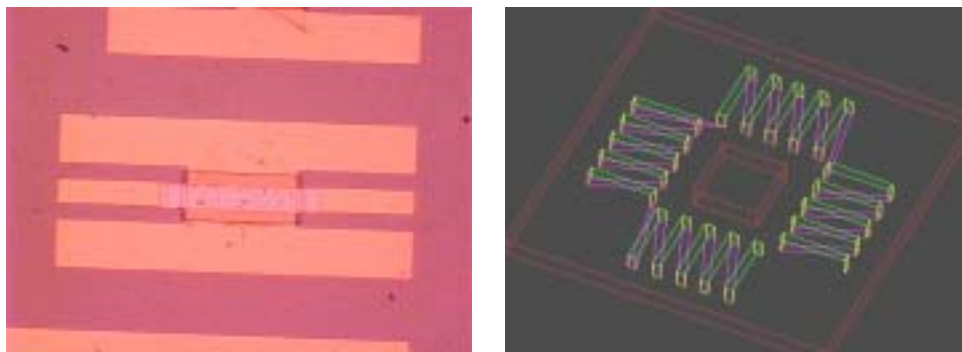
We have also made progress on the design of the high current power supply. We have designed a fluidic oscillator to take the continuous stream of pressure and create an AC fluid stream and we have designed the mechanical to electrical transducer (cantilevered permalloy with multiplayer coil) for converting the pressure stream into electrons. Fabrication and test of the system is impending.



***Figure 6. Cantilevered mechanical to electric generator design.  
Permalloy deposit in red, multilayered coil construction in orange***

### *MicroTemp and Microconductivity*

We have continued investigating thin-film capacitive-type zinc-oxide (ZnO) temperature sensors and coupled-toroidal conductivity sensors for integration on the chemical chips. The ZnO sensor designs are coplanar waveguide (CPW) implementations patterned on high-resistivity silicon (14-mil thick) with sputtered films of ZnO and SiN (as an encapsulant). The conductivity sensors are formed of coupled toroidal inductors built into multi-layer PCB substrates. At present we are investigating improved deposition methods to enhance the pyroelectric response of the ZnO films and are performing full-wave 3D simulations of the toroidal inductors.



***Figure 7. (Left) ZnO temperature-sensitive capacitor integrated in coplanar waveguide on high-resistivity silicon (Right) CAD layout for a 3-D Toroidal inductor built in a multi-layer PCB architecture.***

We have in concert with the physical sensor developments on the chemical chip have made some exploratory progress on commercial off the shelf (COTS) MEMS sensors to create an integrated meteorological sensor suite that uses available sensors. The microphysical transducers are more mature than chemical sensors and thus are commercially available. A proposed in-water prototype instrument containing MEMS P,T, Flow, and Motion sensors has been articulated and we have a lab prototype of P, T and flow including the DAQ system and control loops for future actuation of any carrier.

## **RESULTS**

We have contributed to advances in the field of Maritime MEMS and the techniques needed to fabricate microfluidic-based systems for field analytical purposes. We have an operational LIF-electrophoretic chip instrument and have established the capability for separations based electrochemical detection. We have data on a new type of miniature pump and power source technology. We have progressed seawater sample preparation and analysis for chemicals and have made steps toward a fieldable integrated MEMS physical sensor suite. This project is, as in the past, subject to the time consuming task of method analysis for controlling chemical behavior. Our current focus is to push integration of the lab prototypes with needed mini components and field the system most ready for insertion into the water.

## **IMPACT/APPLICATIONS**

This proof of technology demonstration has impact for both marine science and ocean systems applications as a design and development paradigm that can allow the creation of inexpensive microsensors. Ocean Observing Systems and Operational Oceanography will benefit from the new manufacturing and technology approach.

## **TRANSITIONS**

The work described herein on the microsensor development is a new technological strategy for ocean instrumentation development. We expect this technology practice to emerge as the global ocean observing efforts emerge and large sensor arrays and sensor grids are implemented.

## **RELATED PROJECTS**

We are involved in another (SMDC-ARMY) project aimed at a field sensor for terrestrial monitoring applications.

## **PATENTS, PUBLICATIONS, TECHNOLOGY TRANSFER**

1. Fries, D.P. Langebrake L., Steele, C., Marine-Based Fieldable Microsystems: Emerging and Novel Applications. *Sensors Expo Proceedings*, Chicago IL, June 2001, 201-208.
2. T. Ketterl\*, T. Weller\*, D. Fries\*\*, A Micromachined Tunable Co-Planar-Waveguide Resonator  
\*University of South Florida, Tampa, USA, \*\*IEEE *International Microwave Symposium*, June 2001,

3. Technology Transfer: Maskless Lithography – Intelligent MicroPatterning LLC.
4. Technology Transfer: Giant Magnetoresistance Based Accelerometer – General Dynamics Corp (in process)
5. Patent: Integrated Wireless Motion Sensor using Micromechanical RF Components – Applied for (Weller, Fries, Ketterl)